Kellogg/RL 55-442

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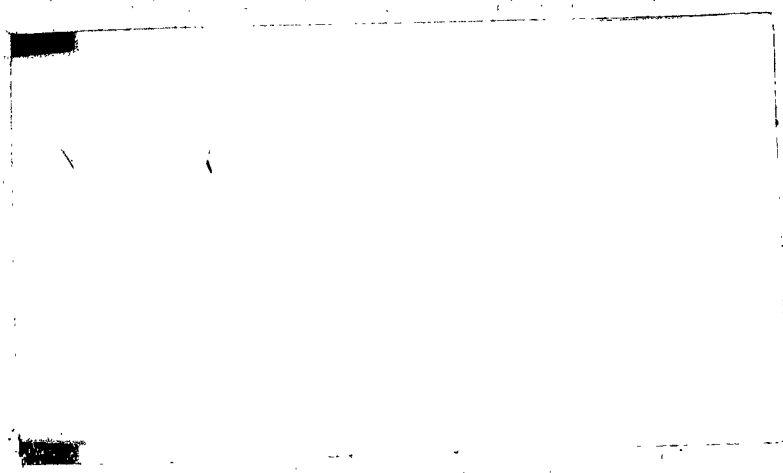


THE M.W.KELLOGG COMPANY

Petroleum and Chemical Research Department

PROGRESS REPORT

ARCTIC RUBBER

U.S. Army Contract DA-44-109-qm-1580
For the Period August - September 1955

December 1, 1955

Copy No. 15 Report RL-55-442

Petroleum and Chemical Research Department
Laboratory Division, Jersey City, N.J.

PROGRESS REPORT

Subject: Arctic Rubber - U.S. Army Contract DA-44-109-qm-1580 for the period August - September, 1955

Staff: J.W. Copenhaver, B.F. Landrum, E.S. Lo, A.N. Bolstad, A.G. Davis, K.S. Tenney

Author: E.S. Lo

Period Covered: August - September, 1955

L.O. No. D-221; Job No. 5675

Previous Reports on this Subject:

RL-50-139	dated	November 1, 1950
RL-51-146	"	February 1, 1951
RL-51-156	"	April 1, 1951
RL-51-163	"	July 1, 1951
RL-51-174	"	October 1, 1951
RL-52-183	"	February 1, 1952
RL-52-195	"	May 1, 1952
RL-52-209	"	August 1, 1952
RL-52-248	"	October 1, 1952
RL-53-259	"	January 1, 1953
RL-53-274	"	April 1, 1953
RL-53-289	"	August 1, 1953
RL-54-329	"	July 29, 1954
RL-54-333	"	August 4, 1954 (Final Sum. Report- DA-44-109-qm-222)
RL-54-353	"	September 30, 1954
RL-54-367	"	December 1, 1954
RL-55-401	"	April 1, 1955
RL-55-422	"	July 1, 1955
RL-55-434	"	September 1, 1955

E. F. SCHWARZENBEK

CONTENTS

I. Introduction

A. Purpose of the Project

The preparation of an oil and fuel-resistant rubber which retains its elastic properties over the range -70°F. to 160°F.; the development of a rubber suitable for use at 500°F.; the investigation and solution of the accompanying problems of monomer preparation, polymerization techniques, and polymer evaluation.

B. Research Program

To achieve this purpose, the Quartermaster Corps. has authorized the M. W. Kellogg Company to conduct a broad investigation of fluorine-containing polymers, which involves monomer synthesis, polymer preparation, and polymer testing.

Many of the monomers desired for investigation are unavailable commercially. In a few cases, these have been synthesized at M. W. Kellogg. Otherwise, the monomers or their precursors are prepared by Dr. Paul Tarrant of the University of Florida, and Dr. Aldrich Syverson of Ohio State University, or obtained on an exchange basis from the Minnesota Mining and Manufacturing Co. and the Polaroid Corporation.

Polymer preparation has received chief emphasis at M. W. Kellogg. The initial phase of this work is the exploratory copolymerization of each new monomer with selected monomers on hand. The results of screening tests on polymers so obtained are used in the selection of new monomer structures, more suitable monomer combinations and mole ratios, and better recipes and polymerization conditions.

Polymer systems exhibiting solvent swell resistance and low temperature characteristics comparable or superior to the chlorotrifluoroethylene-vinylidene fluoride copolymer originally developed on this project are investigated in greater detail. The more outstanding of these will be prepared in pound batches for a more thorough evaluation.

Polymer compounding, testing, and evaluation are conducted by Mr. C. B. Griffis, Angus Wilson, and staff at the Quartermaster Research and Development center at Natick, Mass. ASTM procedures D-471-52T (solvent swell), and D-1053-52T (Gehman Stiffness) are employed in screening the specimens obtained in the exploratory copolymerizations.

C. Past Progress

The copolymer systems investigated were 660, and the rubberlike systems, 332. (Refer to RL-55-434)

II. Summary of Current Progress

The number of monomers available for copolymerization is 84; the number of different polymer systems investigated, 686, and the number of rubberlike systems, 348.

The following monomers have been copolymerized with selected monomers now available: $CF_2=CHCH=CH_2$, $CF_2=CFCF_2Cl$ and vinyl pyridines.

The development of a new high temperature rubber $CF_2=CF_2/CF_2=CFCF_2Cl$ is in progress. The most promising copolymer systems remain to be $CF_2=CH_2/CF_3CF=CF_2$ and $CF_2=CH_2/CF_2=CF_2$.

III. Experimental Section

A. Total Monomers

Eighty-four monomers are now available for copolymerization study. (Refer to RL-55-434).

B. Chemicals Received

The following samples were received during the current period from the Ohio State University:

Compound	b.p., °C.	Amount, g.
$CH_2=CHOCF_2CHClF$	73-74/atm.	
$CHCl_2CHClOCF_2CHClF$	86.5-87.5/30 mm.	
$CH_2ClCHClOCF_2CHClF$	71-73/30 mm.	25
$CH_2CLCH_2OCF_2CHClF$	80-82/100 mm.	
$CF_3CCl_2OCF_2CF_2Cl$	90.5-90.7/741 mm.	
$CH_3CF=CH_2$	-22 to -21.5	1870

The following pyridines were purchased from Reilly Tar & Chemical Corp.:

2-vinyl pyridine (monomer 83) 79-82°/24 mm. 1 lb.
4- " " (" 84) 70-74°/15 mm. 1 lb.

C. Monomer Purification

The crude $CF_2=CFCF_2Cl$ (monomer 82) obtained by the decarboxylation of the sodium salt of C_4 telomer acid was fractionated. The major fraction (ca. 630 g.) boiled between 7-8.5°C., was collected. The mass spectrometric analysis indicates the monomer to be pure.

2 and 4 vinyl pyridine (monomers 83 and 84) were each fractionated under vacuum in order to remove inhibitor and impurities. The water-white purified monomers were stored under N_2 at -70°C.

D. Monomer Analysis

Mass spectrometric analyses of the three fluorinated butadienes received from Dr. Tarrant are as follows:

1. $CF_2=CHCH=CH_2$ (monomer 37)

	Mole % (app.)
$CF_2=CHCH=CH_2$	~70
$C_4H_5F_3$	~22
C_5H_{10}	~ 8
$C_4H_4F_4$	trace

2. $CF_2=CFCH=CH_2$ (monomer 56)

This sample appears to be pure. (Water vapor is the only impurity noted).

3. $CF_2=CHCF=CH_2$ (monomer 51)

	Mole % (app.)
$CF_2=CHCF=CH_2$	~85
$C_4H_4F_4$	~15

E. Polymer Preparation

Polymerization using 686 monomer systems has been attempted. The 26 new systems are: 1-82, 1-84, 2-10, 2-14-72, 2-84, 16-51-56, 16-51-74, 16-56-74, 21-32, 24-84, 37-42, 37-56, 37-72, 37-73, 37-74, 37-84, 51-82, 51-84, 56-82, 56-84, 74-82, 74-83, 74-84, 82, 82-84, and 84.

Of the systems investigated 348 can be considered rubberlike. The 16 new systems are listed below: 2-14-72, 16-51-56, 16-51-74, 16-56-74, 21-32, 24-84, 37-42, 37-56, 37-72, 37-73, 37-74, 51-82, 56-82, 74-82, 74-84, and 82-84.

Experimental data relative to the exploratory work carried out during the current period are set forth below:

1. Homopolymerization of Halogenated Propenes

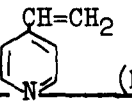
A few attempts to homopolymerize $CF_3CF=CF_2$, $CF_3CCl=CF_2$ and $CF_2=CFCF_2Cl$ (monomers 14, 32 and 82 respectively) in both solution and emulsion recipes failed to give high molecular weight polymers (see runs 3007-8, 3021-22, 3053 and 3060, Table I). In some cases small amounts of yellow oil or powder were obtained.

2. Copolymers of $CF_2=CHCH=CH_2$ (Monomer 37)

This monomer gives a powdery homopolymer. It copolymerizes with fluorinated dienes and propenes to give short rubbery products in good yields (see runs 3009-11, 3023-30, 3036-43, Table I).

3. Copolymers of $CF_2=CFCF_2Cl$ (Monomer 82)

This monomer does not homopolymerize in the regular emulsion recipe (see run 3053, Table I). However, it copolymerized with various fluorinated dienes and ethylenes giving rubbery polymers in good yields (see runs 3054-58, Table I and Table III).

4. Copolymers of  (Monomer 84)

This monomer gives a plastic homopolymer. It copolymerizes with fluorinated dienes, propenes and ethylenes giving colored resinous or stiff rubbery polymers (see runs 3012-23 and 3058, Table I) in good yields.

5. Exploratory Terpolymers

The Gehman T_5 value of the copolymer system $CF_2=CHCF=CH_2$/ $CF_2=CFCH=CH_2$ was lowered six degrees by terpolymerizing the fluorinated dienes with a third monomer $CH_2=CHOCF_2CF_2H$ (see run 1979, Table II). A series of terpolymers (see runs 3035, 3044-47, Table I) have been made during this current period. They will be sent to QM for evaluation as soon as the analytical data are complete.

F. Polymer Evaluation

During the past two months the data of the low temperature flexibility and fuel resistant properties of 25 rubbery polymers were received from the Quartermaster Corps (see Table II).

Among the 25 copolymers, the following five samples: 1-51, 14-51, 18-51, 51-56 and 51-73 (see runs 1959, 1971, 1977, 1984 and 1999, Table II) have volume swells and torsional moduli comparable with X-300 Elastomer, but have better Gehman T_5 values. Different molar ratios of these copolymer systems will be prepared.

IV. High Temperature Rubber Program

The excellent thermal stability, fuel and acid resistance of the $CF_2=CH_2/CF_3CF=CF_2$ copolymers (see Table VIII, RL-55-434) have stimulated the copolymerization of other fluorinated propenes and butenes with vinylidene fluoride or other fluorinated ethylenes.

$CF_2=CFCF_2Cl$ (monomer 82) copolymerizes with $CF_2=CH_2$ to give rubbery products in good yield. The results of a few physical and chemical tests are summarized in Table III. Its resistance to "Esso Turbo Oil 15", the diester type hydraulic fluid, is poor. The tests for thermal stability and fuel resistance are now in progress, and will be reported when the data are available.

V. Plans for Future Work

1. Exploratory polymerizations of new monomers will continue with selected monomers, based upon past experiences.

2. Some theoretical work on the development of new monomer structures and methods of monomer synthesis is planned.

3. The reactivity ratios of $CF_2=CHCF=CH_2$ and $CF_2=CFCH=CH_2$ will be determined.

4. One pound batches of the appropriate molar ratios of the following systems: $CF_2=CHCF=CH_2/CF_2=CFCH=CH_2$, $CF_2=CH_2/CF_3CF=CF_2$ and $CF_2=CH_2/CF_2=CF_2$ will be prepared for evaluation of cured samples.

5. Preparation of ether-linked fluorocarbon polymers will be attempted.

Elizabeth S. Lo

E. S. LO

References to Original Records

Notebook No. 339, pp. 167-189 incl.
" " 308, pp. 176-180 "

THE M. W. KELLOGG COMPANY
Petroleum & Chemical
Research Dept.
Jersey City, N. J.

Report No. RL-55-442

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TABLE I

EXPLORATORY POLYMERIZATION

No.	Comonomers	Mole Ratio Charged /10/	Polymerization Conditions: Time hrs.	Temp. °C.	Recipe	% Conv.	Appearance of Sample /11/: Before Milling	After Milling
2097	$CF_2=CFCl$	100	22	↑	/1/	41	Powder	-
2098	"	100	22	|	/2/	23	Powder	-
2099	"	100	22	|	/3/	80	Powder	-
3000	$CH_2=CFCH=CH_2$	100	22	|	/1/	96	Short Rubber	Short Rubber
3001	$CH_2=CFCH=CH_2$/$CF_2=CFCH=CH_2$	50/50	22	|	/1/	84	"	"
3002	$CF_3CCl=CH_2$/$CH_2=CFCH=CH_2$	50/50	72	|	/3/	45	"	"
3003	" /$CH_2=C(CF_3)CH=CH_2$	50/50	72	|	/3/	28	Soft Resin	-
3004	" /$CH_2=CFCl$	50/50	72	|	/3/	1	Low Mol.Wt. Polymer	-
3007	$CF_3CF=CF_2$	100	68	50	/4/	0.3	Powder	-
3021	"	100	48	|	/5/	1	Powder	-
3008	$CF_3CCl=CF_2$	100	68	|	/4/	0.5	Powder	-
3022	"	100	48	|	/5/	zero	-	-
3009	$CF_2=CHCH=CH_2$/$CH_2=CHOCF_2CF_2H$	80/20	68	|	/3/	54	Short rubber	Short rubber
3010	" /$CF_3CF=CF_2$	70/30	68	|	/3/	44	Powder	-
3011	" /$CF_3CCl=CF_2$	70/30	68	|	/3/	40	Rubbery powder	Short rubber
3023	" /$CF_3CH=CH_2$	70/30	25	|	/6/	58	Short rubber	Smooth short rubber
3024	" /$CF_3CH=CF_2$	70/30	25	|	/6/	48	Rubbery powder	Smooth short rubber
3025	$CF_2=CHCH=CH_2$/$Cl(CF_2CFCl)_2CF_2CO_2$-$CH_2CH=CH_2$	90/10	25	|	/6/	60	"	"
3026	" /$CF_2=CHCF=CH_2$	90/10	70	25	/6/	68	"	Very short rubber
3027	" / "	80/20	70	25	/6/	62	Short rubber	Tough short rubber
3028	" / "	50/50	70	25	/6/	92	"	"
3029	" / "	25/75	70	25	/6/	90	Tough short rubber	Rubbery sheet
3030	$CF_2=CHCH=CH_2$	100	70	25	/3/	40	Powder	-
3012	 / $CF_2=CHCH=CH_2$	50/50	20	40	/3/	50	Yellow flakes	-
3013	" /$CF_2CHCF=CH_2$	50/50	20	40	/3/	26	Brown flakes	-
3014	" /$CF_2=CFCH=CH_2$	50/50	20	40	/3/	83	Brown flakes	-
3015	" /$CH_2=CHC(CF_3)=CH_2$	50/50	20	40	/3/	23	Soft yellow rubber	-
3016	 homopolymer	100/0	24	25	/3/	64	Yellow plastic	-
3017	" /$CH_2=CF_2$	40/60	24	25	/3/	40	Lt. yellow plastic	-
3018	" /$CF_2=CFCl$	50/50	24	25	/3/	80	Black flakes	-
3019	" /$CF_2=CF_2$	45/65	24	25	/3/	43	Yellow chips	-
3020	" /$CH_2=CHC(CF_3)=CH_2$	5/95	60	25	/3/	92	Soft rubber	Weak crepe sheet
3021	" / "	15/85	60	25	/3/	92	Short stiff rubber	Stiff crepe sheet
3023	" / "	5/95	60	25	/3/	92	Soft rubber	Crepe sheet
3022	 $CH_2=CH_2$/$CH_2=CHC(CF_3)=CH_2$	5/95	60	25	/3/	96	Soft sticky rubber	Weak sticky sheet
3036	$CF_2=CHCH=CH_2$/$CF_2=C(CH_3)CH=CH_2$	75/25	40	25-40	/3/	8	Soft rubber	Short snappy rubber
3037	" /$CF_2=CFCH=CH_2$	90/10	40	25-40	/3/	51	Short stiff rubber	Tough stiff sheet
3038	" / "	80/20	40	25-40	/3/	64	Short stiff rubber	Tough stiff sheet
3039	" / "	50/50	40	25-40	/3/	70	"	Tough flexible sheet
3040	" / "	40/60	40	25-40	/3/	80	Short tough rubber	Tough stiff sheet
3041	" /$CH_2=C(CF_3)CH=CH_2$	75-25	40	25-40	/3/	51	Soft rubber	Soft, translucent rubber
3042	" / "	50/50	40	25-40	/3/	65	Soft rubber	Soft, snappy rubber
3043	" / "	25/75	40	25-40	/3/	75	Soft, weak rubber	Soft, translucent rubber
3034	$CF_2=CH_2$/$CF_3CF=CF_2$	95/5	18-1/2	50	/6/	80	Rubbery powder	Plastic
3048	$CF_2=CFH$/$CF_3-C(Cl)=CF_2$	70/30	24	50	/6/	47	Sl. rubbery particles	Flexible plastic sheet
3049	" / "	60/40	24	50	/6/	60	Sl. rubbery crumbs	"
3052	$CF_2=CH_2$/$CH_3CH=CH_2$	70/30	22	50	/6/	zero	-	-
3053	$CF_2=CFCF_2Cl$	100/0	24	50	/6/	zero	-	-
3054	$CF_2=CFCF_2Cl$/$CF_2=CHCF=CH_2$	30/70	24	50	/6/	28	Rubber	Snappy rubber
3055	" /$CF_2=CFCH=CH_2$	30/70	24	50	/6/	58	Hard short rubber	Very short rubber
3056	" /$CH_2=C(CF_3)CH=CH_2$	30/70	24	50	/6/	51	Snappy rubber	Soft rubber
3057	" /$CF_2=CFCl$	30/70	24	50	/6/	34	White powder	-
3058	" / 	30/70	24	50	/7/	50	Brown short rubber	-
3060	$CF_3CCl=CF_2$	100/0	66	25	/8/	low	Yellow oil	-
	Terpolymers							
3035	$CF_2=CH_2$/$CF_3CF=CF_2$/$CH_2=CHOCF_2CF_2H$	80/10/10	18-1/2	50	/6/	76	Very short rubber	Tough leathery sheet
3044	" / " / "	60/36/4	17-1/2	50	/6/	73	Soft rubber	Soft rubber
3045	$CF_2=CHCF=CH_2$/$CH_2=C(CF_3)CH=CH_2$/$CH_2=CHCH$	50/40/10	24	50	/9/	82	Soft rubber	Crepe rubber
3046	" /$CF_2=CFCH=CH_2$/$CH_2=CHCH$	40/50/10	24	50	/6/	90	Sl. short rubber	Crepe short rubber
3047	$CF_2=CFCH=CH_2$/$CH_2=C(CF_3)CH=CH_2$/ "	50/40/10	24	50	/6/	92	Tough rubber	Crepe short rubber

/1/ Recipe: Water 200; Perfluorooctanoic acid 0.75; $K_2S_2O_8$ 1; $Na_2S_2O_5$ 0.4; monomer 100; n-hexane 1.32; pH of polymerization medium 7.

/2/ Recipe: Same as recipe /1/, except n-hexane 2.64.

/3/ Recipe: Same as recipe /1/, except no n-hexane.

/4/ Recipe: Acetone 200; benzoyl peroxide 1; monomer 100.

/5/ Recipe: Tetrahydrofuran 200; benzoyl peroxide 1; monomer 100.

/6/ Recipe: Water 200; perfluorooctanoic acid 1; $K_2S_2O_8$ 1; $Na_2HPO_4 \cdot 7H_2O$ 4; monomer 100.

/7/ Recipe: Water 200; NH4R 5; $K_2S_2O_8$ 1; $Na_2S_2O_5$ 0.4; pH of polymerization medium 7.

/8/ Recipe: $CF_3CCl=CF_2$ 8.5 g.; From 114 5 cc.; HF_3 0.12 g. (ca 45 cc.); Sealed in stainless steel bomb and stood for 66 hrs.

/9/ Recipe: Water 200; Perfluorooctanoic acid 1; $K_2S_2O_8$ 1; $Na_2S_2O_5$ 0.4; pH of polymerization medium 7.

/10/ Recipe: The combined molar ratios of these polymers will be reported when the analytical data are available.

/11/ Recipe: All the samples banded on mill at 25°C., except runs 3024, 3025 and 3026 which banded at 80°C.

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TABLE II

ARCTIC RUBBER SCREENING TEST

Run No.	Monomer Structure	Molar Ratio Charged	Molar Ratio Combined	% Conv.	Appearance of Sample Before Milling	Appearance of Sample After Milling /1/	Gehman Stiffness (°C.) T_2	T_5	T_{10}	T_{100}	Vol. of Increase Type II Fuel	Torsional Modulus (psi)	Molding Temp. (°F.) /2/	Sample Condition After Molding
	Copolymers													
1999	$CF_2=CFCl/CF_2=CHCF=CH_2$	50/50	13/87	49	Rubber	-	-16.4	-24.0	-27.6	-37.6	15	372	30/250; 250, 200	Firm & rubbery
1971	$CF_3CF=CF_2/CF_2=CHCF=CH_2$	25/75	Loss of F	43	"	Short rubber	-12.9	-18.8	-23.3	-37.8	9	372	30/250; 30/110	Stiff & rubbery
1972	" $/CH_2=C(CF_3)CH=CH_2$	25/75	Loss of F	64	"	Soft rubber	+19.8	- 1.0	-11.7	-24.7	Gel	5	Cold mold	Soft & gummy
1973	$CH_2=CHCN/$ "	25/75	20/80	95	"	Slightly tough rubber	+ 9.0	- 7.8	-15.3	-28.9	27	136	250	Firm & plastic
1959	$CF_2=CHCl/CF_2=CHCF=CH_2$	10/90	1.5/98.5	62	"	Slightly short rubber	-11.3	-20.6	-23.8	-33.0	12	222	250	Stiff & rubbery
1960	$CF_2=CHCl/$ "	25/75	2/98	50	"	"	-15.4	-21.1	-24.7	-36.1	10	589	250	Stiff & plastic
1961	$CF_3CCl=CH_2/$ "	10/90	11/89	66	Snappy rubber	Crepe rubbery sheet	+ 4.2	-10.3	-14.3	-24.9	15	24	30/250	Soft & rubbery
2001	$CF_2=CHCF=CH_2/CF_2=CFCH=CH_2$	95/5	-	87	Short rubber	Soft rubber	-14.1	-24.1	-27.1	-42.9	7	988	250	Stiff & plastic
2000	" / "	90/10	-	84	"	"	-15.1	-24.3	-27.3	-39.6	11	637	250	Stiff & plastic
1984	" / "	80/20	-	70	Rubber	Crepe rubbery sheet	-15.2	-23.5	-25.5	-33.8	20	191	250	Firm & rubbery
1983	" / "	75/25	-	84	Rubber	"	-20.4	-24.4	-27.1	-37.0	24	120	250	"
1982	" / "	60/40	-	78	"	"	- 6.7	-23.9	-24.5	-34.3	37	71	250	"
1986	" / "	50/50	-	76	"	Tough crepe rubbery sheet	-17.4	-26.9	-29.1	-39.8	40	133	250	Stiff & rubbery
1985	" / "	40/60	-	80	"	"	-15.3	-27.3	-29.5	-39.7	37	504	250	"
1976	" $/Cl(CF_2CFCl)_2CF_2CO_2CH_2CH=CH_2$	79/21	95.5/4.5	35	"	Rubbery crepe sheet	- 8.0	-14.3	-17.0	-26.3	67	135	250	"
1977	" / "	90/10	97.5/2.5	49	"	"	-11.5	-20.0	-23.1	-33.8	4	464	250	"
2002	$CF_2=CFCH=CH_2/CH_2=C(CF_3)CH=CH_2$	80/20	69/31 /3/	99	"	Soft rubber	-26.8	-30.7	-32.8	-41.7	80	126	250	Firm & rubbery
2003	" / "	75/25	65/35 /3/	91	"	"	-23.8	-29.3	-32.0	-40.7	85	130	250	"
2004	" / "	60/40	15/85 /3/	99	"	"	- 2.8	-19.3	-24.5	-33.4	96	34	250	Soft & rubbery
	Terpolymers													
1978	$CF_2=CHCF=CH_2/CF_2=CFCH=CH_2/CF_3CCl=CH_2$	50/40/10	-	72	Rubber	Rubbery crepe sheet	-15.3	-22.5	-23.9	-31.8	43	62	250	Soft & rubbery
1979	" / " $/CH_2=CHOCF_2CF_2H$	50/40/10	-	56	Soft rubber	Short rubber	-26.7	-32.5	-34.2	-39.6	46	289	250	Stiff & rubbery
1980	" / " $/Cl(CF_2CFCl)_2CF_2CO_2CH_2CH=CH_2$	"	-	60	Rubber	Slightly tough rubber	-15.6	-22.3	-25.6	-34.3	40	72	250	"
1981	" / " $/CH_2=C(CF_3)CH=CH_2$	50/40/10	-	78	Rubber	Rubbery crepe sheet	-20.9	-25.4	-27.3	-37.0	48	40	250	Firm & rubbery
1964	" $/CH_2=C(CF_3)CH=CH_2/CF_2=CHCl$	40/40/20	-	74	Soft rubber	Soft rubber	-20.5	-27.2	-29.2	-35.0	83	263	30/200, 250	Soft & rubbery
1963	" / " $/CF_3CCl=CH_2$	40/50/10	-	62	Rubber	Rubbery crepe sheet	-17.0	-23.4	-25.9	-33.9	92	196	20/110	Soft & gummy

/1/ All the samples banded on mill at 25°C.

/2/ Mold times unless otherwise noted are 10 minutes. Longer periods are totals of individual 10 minute periods.

/3/ Reason for unexpected analytical results not determined.

TABLE III

COPOLYMERS OF $CF_2=CH_2/CF_2=CFCF_2Cl$

Run No.	3050	3051	3059	3061
Molar Ratio of $CF_2=CH_2/CF_2=CFCF_2Cl$:				
Charged	70/30	60/40	50/50	25/75
Combined	75.5/24.5	68.4/31.6	64.8/35.2	55.2/44.8
Polymerization Conditions				
Time, hrs.	22-1/2	22	69	23
Recipe	/1/	/1/	/1/	/1/
% Conversion	88	77.5	66.7	34
Appearance of Sample:				
Before milling	White Rubber	White Rubber	White Rubber	Sl. hard Rubber
After milling /2/	Crepe rubbery sheet	Crepe rubbery sheet	Crepe rubbery sheet	Crepe rubbery sheet
Torsional Modulus, psi	180	169	168	178
Gehman values, °C. /3/				
T_2	+3	+9	+11	+13
T_5	-3	+3	+5	+10
T_{10}	-6	0	+3	+9
T_{100}	-16	-9	-6	+4
Resilience (Bayshore)	7	10	5	-
"Esso Turbo Oil 15"				
18 hrs. at 77°F. wt. % increase	35	19	47.8	-
1 hr. at 600°F.	Partially soluble			-

/1/ Recipe: Water 150; C_8 Telomer Acid 0.75; $Na_2HPO_4 \cdot 7H_2O$ 3; $K_2S_2O_8$ 0.75; Monomer 50-60

/2/ All samples banded on mill at 25°C.

/3/ All samples were molded at 300°F.

FILE NO. MM-1403
ESL:MM 9-30-55